

# Status of Research on Deposition of Thin Superconducting Films for RF Accelerating Cavities<sup>1</sup>

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**Abstract –** In 2000 the ultra-high vacuum (UHV) arc-technology was proposed as an alternative method for depositing thin superconducting films of pure niobium upon inner surfaces of RF cavities designed for particle accelerators. It is well known that the cathodic arc deposition technology offers an excellent approach to produce pure metal, alloy- and compound-films with excellent adhesion and densities, at relatively high rates. Higher ion energies are the main factor allowing produce more compact films, with much stronger adhesion than those obtained by means of other methods. It was shown that the cathodic arc working at UHV conditions solves the problem of the oxygen contamination coming from a water vapor, and this opens the road to applications where very pure metallic films are needed. This paper describes the status of research on the deposition of superconducting films upon RF accelerating cavities. The main results and characteristics of the arc-deposited thin superconducting niobium films, as well as the progress obtained recently in the formation of such films, are presented.

## 1. Introduction

Vacuum arc technology is widely used in scientific laboratories and industry in order to produce different coatings upon various surfaces. The deposition of pure and clean metal films of different types, as well as possibility of the vacuum arc operation within a reactive-gas environment (in order to form compound films, such as nitrides, oxides and carbonaceous layers), make this technology very attractive. Appropriate surface layers can be formed upon constructional parts of complicated shapes, ensuring high bonding strength and higher corrosion resistance. In a comparison with other deposition techniques, e.g. the known Physical Vapor Deposition (PVD) process, when ions have energy of a few eV, vacuum arc discharges can produce ions of higher kinetic energies ranging from about 15 eV to about 150 eV [1]. It results in the formation of a denser film and it strongly reduces surface defects, such as voids and

columnar growth. The main drawback is the production of micro-droplets (macro-particles) in the near-cathode region. Such micro-droplets are ejected away from the cathode region and they become embedded in the film, what increases its surface roughness. In order to eliminate the micro-droplets from vacuum-arc plasmas, one can apply different magnetic filters. The main idea is to transport a plasma stream by means of an appropriate magnetic field and to eliminate micro-droplets, which (due to their large masses) move from the cathode along almost straight lines.

In connection with different R&D programs concerning the construction of large linear accelerators, particular attention was paid to possibilities of the deposition of thin super-conducting layers, e.g. by the magnetron sputtering [2]. The adhesion of such layers to surfaces of the accelerator resonance cavities appeared to be not very strong. Therefore, a new concept of the deposition of thin super-conducting layers by arc discharges under Ultra-High Vacuum (UHV) conditions was proposed several years ago [3, 4]. A considerable progress in the development of this technology has been achieved recently, including a problem of the effective elimination of micro-droplets in planar-arc facilities. Our previous report [5] described concepts of 2 types of the magnetic filters suited for UHV conditions. Some experimental research on them is still under realization, particularly in order to improve efficiency of the micro-droplets filtering and plasma transport through the magnetic channel. In fact the filters with the water cooling system work very stably and their constructions enable the long-lasting operation. Additionally, it is possible to bake out the inner surface of the filter when very clean conditions are required. Another paper [6] was devoted to residual gas measurements in a vacuum chamber at the UHV conditions: before, during and just after the vacuum-arc

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deposition process. Moreover, a comparison of gas compositions between HV and UHV has been presented. Such an analysis is very important when very clean layers are needed, e.g. superconducting niobium films. The technology of the filtered vacuum arc at UHV conditions opens a new road to many applications, when very pure metallic- and/or superconducting-films are needed.

This paper reports on recent progress in the application of the UHV arc technology, which was proposed as an alternative solution for the deposition of thin superconducting films of pure niobium (Nb) upon the inner surfaces of RF cavities designed for particle accelerators. The status of our joint research on the deposition of such superconducting films, designed for RF accelerator cavities of the TESLA type, is described. The main results and characteristics of arc-deposited thin superconducting films are discussed, and the progress achieved recently in the formation of such films is presented.

## 2. Experimental systems

For the coating of the inner surfaces of RF accelerating structures, there were proposed two various approaches. First one has been based on a linear (cylindrical) geometry of the arc source. A cylindrical cathode of the linear-arc can be oriented along the cavity axis. Changing a position of an additional permanent magnet, which might be located inside of the tubular Nb cathode, one can drive the arc discharge along the symmetry axis – in order to obtain uniform coating of a single cavity or multi-cell structure. A scheme of such a system, which has been designed at IPJ, is shown in Fig. 1.

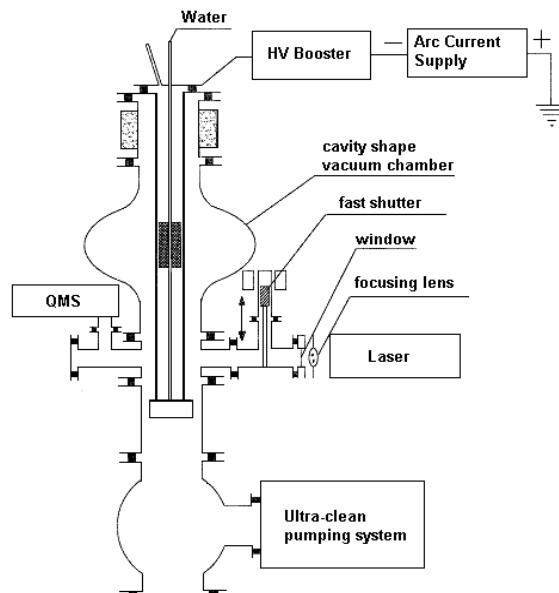


Fig. 1. Schematic drawing of an UHV apparatus with the linear-arc source for the single-cell cavity coating

General view of a linear-arc facility, which was designed and constructed at IPJ in Swierk, is presented in Fig. 2.



Fig. 2. UHV system with a linear-arc and a TESLA-type RF cavity, as shown during tests in Swierk

The second approach has been based on an arc source with a planar (in fact a truncated cone) cathode, which is shown in Fig. 3.

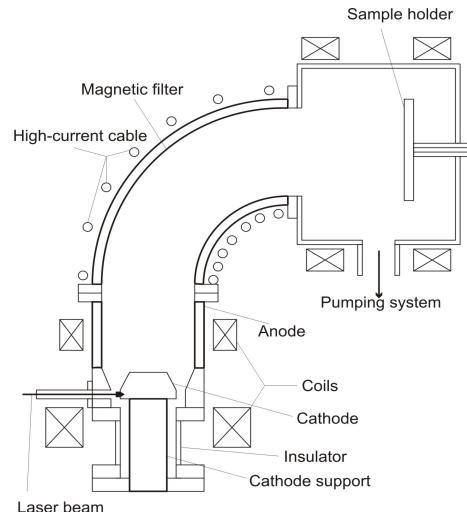


Fig. 3. Scheme of a UHV arc facility with a planar cathode and a knee-type magnetic filter

Such a system consists of a truncated-cone cathode fixed upon a water-cooled support placed inside a vacuum chamber. To reduce an amount of micro-droplets the use is made of a special magnetic filter, which collects the micro-droplets and deflects a plasma-ion stream. Such planar arc sources were constructed both at the Tor Vergata laboratory in Rome and at the IPJ in Swierk. The detailed description of both approaches and their constructions and performance can be found in our previous papers [3–5]. In order to deposit thin Nb layer upon the inner sur-

face of the RF cavity some modifications of the planar facility have recently been proposed. A general view of one of the planar-arc facilities constructed at Tor Vergata laboratory in Rome, and equipped with a magnetic filter, is shown in Fig. 4.



Fig. 4. Planar-arc sources equipped with the focusing coil and a magnetic filter, during tests at Tor Vergata

The described facilities were equipped with oil-free pumping systems, each consisting of a two-stage fore-vacuum pump and a turbo-molecular pump. The achievement of very good vacuum has been of primary importance, because the previous studies showed that thin niobium films, as deposited by arc discharges under UHV conditions, have properties comparable with the pure bulk niobium [7]. In our UHV facilities the basic pressure  $<10^{-11}$  hPa is achieved after 24 hours of baking at 150 °C.

For the arc ignition one must produce a small plasma burst of a sufficient density in order to form a high-conductivity path between cathode and anode, which in our case is generated by a 50-mJ Nd-YAG pulsed laser focused upon the cathode surface. Such a triggering system provides the ultra-clean and reliable ignition.

### 3. Formation of Nb films and studies of their superconducting properties

In our filtered vacuum-arc systems there can be coated several sapphire- and Cu-substrates simultaneously. The samples can be mounted upon a sample holder consisting of a massive Cu (OFHC) flange (see Fig. 3) and kept at a constant temperature during the whole deposition process. The sample holder is electrically insulated from walls of the vacuum chamber, so that a bias of 20–100 V, both in DC and kHz pulse regime, can be applied to the coated substrates. The lowest possible arc current, needed for the stable operation in the applied DC mode, has been found to be about 60 A for, while the available cooling system of the anode has an upper limit of the arc current equal to 140 A. The deposition rate achievable with the system operated at arc currents of 80–100 A is about 1 nm/s. The temperature of the sample during depositions is recorded by means of

thermocouples. Most samples have been deposited at temperatures close to the room temperature, but several samples have been coated at higher temperatures (100–200 °C). The residual pressure in our systems is usually set within the  $10^{-11}$  hPa range. When the arc discharge starts the pressure usually increases to  $10^{-6}$ – $10^{-7}$  hPa and it remains almost stable throughout the whole deposition process. The gas pressure rise during the arc discharge is found to be almost exclusively caused by hydrogen, which partial pressure is usually more than 3 orders of magnitude higher than that of other contaminants [6].

The UHV arc-deposited Nb-layers upon sapphire substrates have been characterized by measuring their critical temperature  $T_c$  and Residual Resistivity Ratio (RRR – defined as the ratio of the resistivity at a room temperature to that measured at 10 K). Both parameters are in general very sensitive to impurities, e.g. very small amounts of oxygen in the Nb-film can lower its  $T_c$  significantly. The RRR values of our 1.5- $\mu\text{m}$ -thick Nb-films, which were deposited upon the sapphire substrates at a room temperature, under the typical UHV conditions, have ranged from 20 up to 50. The record value of RRR=80 was obtained by heating up the substrate to the temperature of 150 °C [7]. The critical temperature ( $T_c$ ) and critical current density ( $J_c$ ) values of the deposited Nb films were measured by means of an inductive method. The best samples have shown the values identical to bulk Nb, i.e.  $T_c=9.26$  K,  $T_c=0.02$  K and  $J_c=3 \cdot 10^7$  A/cm<sup>2</sup>.

### 4. Other characteristics of thin films

Information about the surface chemical composition and depth profile were obtained by means of a time-of-flight (ToF) SIMS mass-spectrometer, produced by the Ion-ToF GmbH, Muenster, Germany. The instrument was equipped with a liquid-metal  $^{69}\text{Ga}^+$  primary-ion gun and ToF mass-analyzer with the high resolution. Depth profiles were obtained by recording secondary ion emission intensity during sequential removing of surface layers from the sample material by ion sputtering, as shown in Fig. 5.

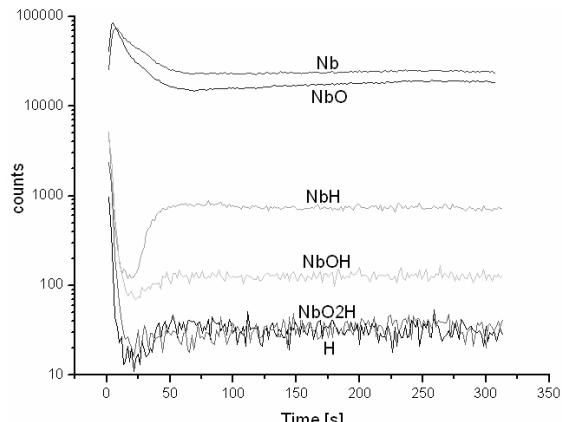


Fig. 5. SIMS analysis of the deposited Nb layer

The SIMS analysis was performed by irradiation of  $300 \times 300 \mu\text{m}^2$  area of the sample surface with 1-keV  $\text{O}_2^+$  ion-beam of 40 nA current. The secondary ion mass-analysis was carried by rastering 25 keV  $^{69}\text{Ga}^+$  primary ion pulsed beam over  $100 \times 100 \mu\text{m}^2$  sample surface. A primary current of 2 pA, a pulse width of ~650 ps and 10 kHz repetition rate were set during that analysis. Secondary ions emitted from the bombarded surface were mass-separated and counted with the ToF analyzer. In Fig. 5 one can easily see that the deposited Nb-layers were clean enough. The deposited layers consisted mainly of pure Nb, however, relatively high levels of NbO, NbH and NbOH species were also observed. The presence of some heavy impurities (like Cs, Na and K species) has also been observed, but their amounts were very low (not shown in Fig. 5). A relatively high level of the oxidation in the near-surface layer is typical for Nb layers.

In order to analyze surfaces of the deposited layers the use was also made of the Scanning Electron Microscopy (SEM), which is a very useful tool to perform the quality inspection for small-scale defects and to look at the surface structure. Some results of the SEM analysis, which was performed at The Warsaw Technical University, are shown in Fig. 6.

Figs. 6, a, b show the SEM pictures of the niobium layers, which were taken with different magnifications. One can see that surface presented on the Fig. 6a is very homogeneous and dense. A lack of micro-droplets upon the surface is the confirmation of the effective plasma filtering, which was performed for the planar arc source by means of a knee-type magnetic filter. Using the higher magnification (Fig. 6, b) it is of course possible to analyze the surface structure. One can see the longitudinal shape of surface grains. The roughness of the arc deposited Nb layers upon sapphire substrates was found to be of the order of few tenth of nanometer. In order to avoid micro-droplets upon the film surface it is necessary to filter the plasma. In our case, the micro-droplets of high-purity molten Nb are not expected to contaminate the film, but they can increase its surface roughness, and within a high RF electric field they may become electron-emitters. An example of such micro-droplets can be seen in Fig. 6, c. The presented picture of the deposited Nb layer shows different micro-droplets upon the investigated surface.

The presence of the micro-droplets upon the deposited films was studied also by means of the optical microscopy. Using an optical microscope with a 500x magnification, there were taken pictures of the sample surface (not shown in the paper) at 10 points chosen randomly. Those pictures have been analyzed using a LabView code, which made possible to record the number and sizes of the micro-droplets visible in the microscope viewing field (fixed area). The surface density of the observed micro-droplets as a function of their sizes, which was measured upon the Nb-coated sapphire sample, is shown in Fig. 7.

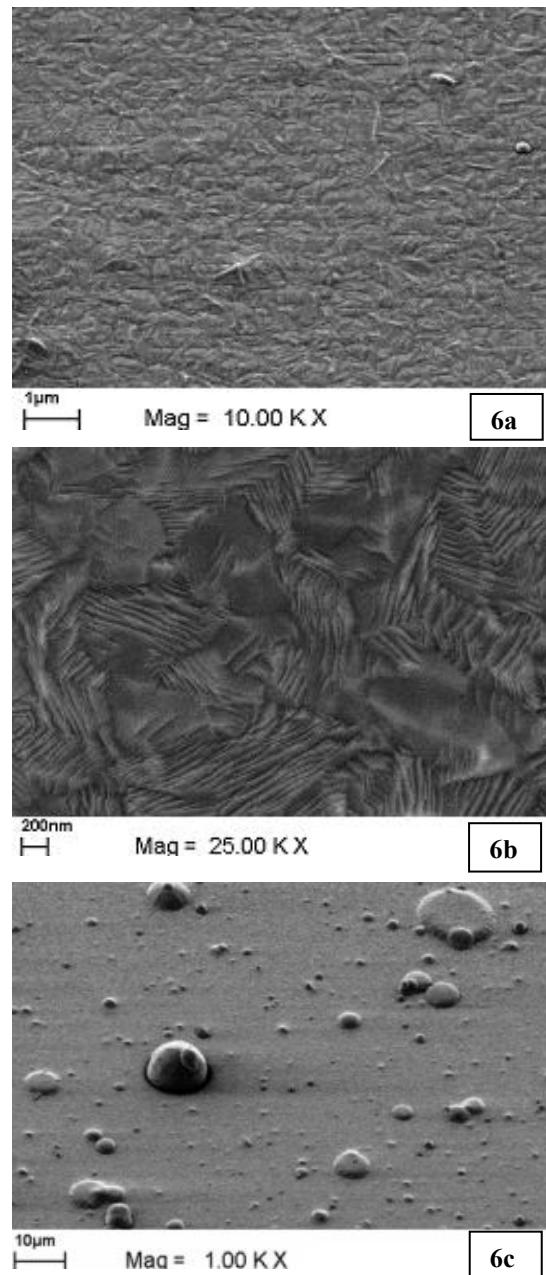


Fig. 6. SEM pictures of the niobium film, which was arc-deposited upon the sapphire substrate. Figs. 6, a and 6, b present the surface coated by means the facility equipped with an UHV planar source and a magnetic filter. Fig. 6, c shows a photo of the surface coated with not-filtered arc plasma

Dimensions of the most observed droplets are comparable in sizes to the Nb film grains (200–300 nm). One could expect that many micro-droplets become embedded in the growing film. Nevertheless, since such macro-particles can increase the surface roughness and possibly porosity as well as the field emission capacity, for our future applications (within high-field RF cavities) they must be eliminated by proper filtering. This can be done by the magnetic deflecting of the arc-produced plasma flow

in such a way that the coated substrate cannot see the cathode surface directly.

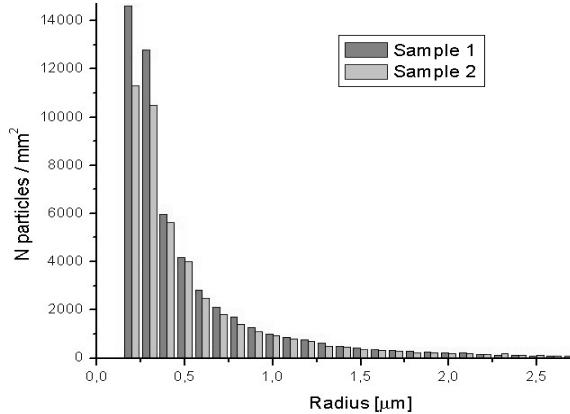


Fig. 7. Typical histogram showing the surface density of the micro-droplets deposited and observed upon surfaces of two samples coated within the facility equipped with a UHV linear (cylindrical) arc-source without any magnetic filter. The investigated samples were coated at different discharge currents: sample 1 – at 55 A, and sample 2 – at 80 A

## 5. Micro-droplets filters

The construction of magnetic filters for the UHV conditions is more difficult than that for the filters operated at higher basic pressures. The main differences are higher requirements as regards the constructional materials and their vacuum behavior, as well as necessity of the application of additional heaters for baking. Moreover, the magnetic filters for their long- and stable-operation must be cooled by an appropriate water flow. As it was mentioned above the magnetic filters suited for the UHV conditions were successfully implemented in planar-arc facilities [5].

For the linear (cylindrical) arc source special constructions of magnetic filters must be developed. Recently, a cylindrical magnetic filter for the elimination of micro-droplets from the arc-produced plasma within the UHV linear-arc facility has been also designed and constructed at IPJ in Swierk. It was assumed that the cylindrical magnetic filter should constitute a tubular set of two cylinders penetrable for arc plasma, but eliminating many micro-droplets. Such cylinders might consist of thin copper tubes distributed symmetrically around the cylindrical surfaces, and supplied by appropriate magnetizing currents.

In order to optimize the configuration of magnetic field lines some model computations have been performed using a 2D Maxwell-type code. Such computations have been carried out for various numbers and diameter of the tubes, as well as for different diameters of the inner- and outer-cylinder. Taking into account the diameter of the central Nb cathode and comparing the computed magnetic fields it was possible to find the optimal configuration. An example of the modeling is shown in Fig. 8.

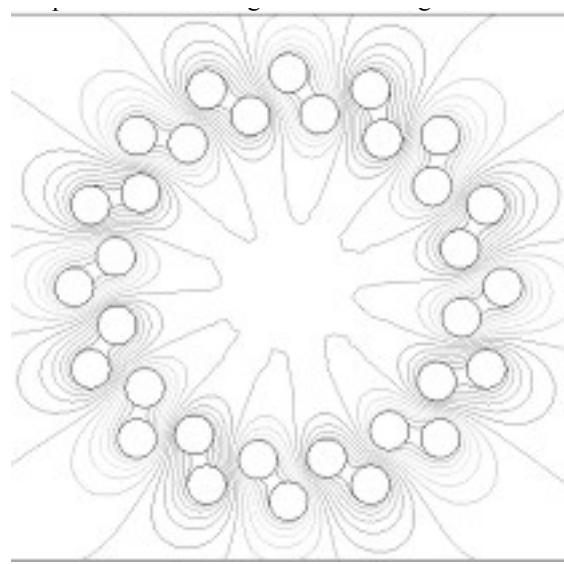


Fig. 8. Modeling of the magnetic field distribution in a cross-section of the chosen cylindrical magnetic filter



Fig. 9. General view of the cylindrical magnetic filter before its installation

The results presented in Fig. 8 were obtained for the set consisting of two coaxial cylinders of 42 mm and 55 mm in diameter, respectively. Each cylinder was composed of 18 water-cooled copper tubes distributed symmetrically (every 20°) around the z-axis. It was assumed that in each cylinder the magnetizing current, passing through the neighbor tubes, flows in the opposite direction. The mutual angular shift between two cylinders was chosen to be equal to

10°. The length of the copper tubes was established to cover the active part of the Nb-cathode, determined by the length of a single TESLA-type RF-cavity. In a future UHV linear-arc facility, which is needed for coating of multi-cell cavities, the length of the individual tubes and the whole filter must correspond to the length of the multi-cell system.

It is evident that, independently on filter dimensions, the tubes must be connected at the ends by electrical connectors, which enable an appropriate magnetizing-current and cooling-water flows to be realized. All the tube connections must of course be vacuum-tight and able to withstand some heating during the arc operation. In order to fix the tubes in proper positions the use might be made of special ceramic-insulator plates and auxiliary supports. A picture of the prototype cylindrical magnetic filter, which has been designed and constructed at IPJ in Swierk, is presented in Fig. 9.

## 6. Concept of RF cavity deposition by means of a planar arc source and cusped field

The deposition of superconducting layers upon the inner surfaces of RF cavities by means of the UHV planar-arcs requires two fundamental problems to be solved. The first problem is related with a transport of the arc-plasma stream from the cathode to the RF cavity. In order to eliminate micro-droplets it is necessary to apply a filter located between the source and cavity, as described above. The plasma

transport can be realized by an appropriate magnetic field, but it usually causes plasma losses and reduction of a deposition rate. The second problem concerns the formation of a uniform coating upon cavity walls.

In order to improve the coating uniformity, a new concept of the deposition has been developed, basing on a so-called "cusp" configuration. For such purpose the use is made of at least two coils with current flowing in the opposite direction. Some magnetic field lines become then inverted and they form the spindle-cusp. The computer modeling of such configurations has been performed at IPJ, and an example of the magnetic field lines distribution inside a multi-cell system is presented in Fig. 10.

The described configuration enables to bend trajectories of plasma ions, which follow the magnetic field lines. Changing intensity of currents flowing through individual coils, one can move a region where the magnetic field lines cross the cavity wall and improve the coating uniformity.

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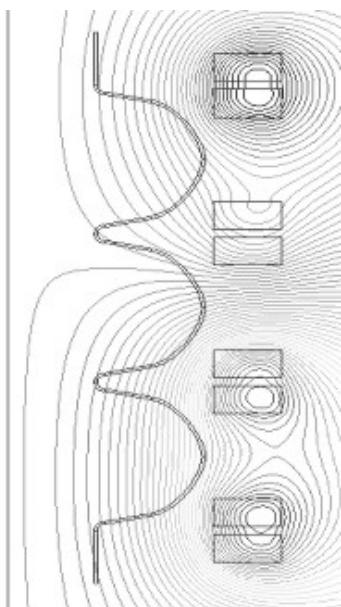


Fig. 10. Computer simulation of the magnetic field lines distribution in the cylindrical "cusp" geometry enabling to improve coating of the inner wall of the multi-cell RF cavity